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(54) **Functionally graded rare earth permanent magnet**

(57) A functionally graded rare earth permanent magnet is in the form of a sintered magnet body having a composition  $R^1_a R^2_b T_c A_d F_e O_f M_g$  wherein the concentration of  $R^2/(R^1+R^2)$  contained in grain boundaries surrounding primary phase grains of  $(R^1, R^2)_2 T_{14} A$  tetragonal system within the sintered magnet body is on the average higher than the concentration of  $R^2/(R^1+R^2)$  contained in the primary phase grains,  $R^2$  is distributed such that its concentration increases on the average from

the center toward the surface of the magnet body, the oxyfluoride of  $(R^1, R^2)$  is present at grain boundaries in a grain boundary region that extends from the magnet body surface to a depth of at least 20  $\mu m$ , and the magnet body includes a surface layer having a higher coercive force than in the interior. The invention provides permanent magnets having improved heat resistance.

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that is, the magnet interior has a distribution of the magnitude of diamagnetic field. For example, if a plate-shaped magnet has a magnetic pole on a wide surface, the center of that surface receives the maximum diamagnetic field. Furthermore, a surface layer of the magnet receives a large diamagnetic field as compared with the interior. Accordingly, when the magnet is exposed to high temperature, demagnetization occurs from the surface layer. Regarding R-Fe-B sintered magnets (wherein R is one or more elements selected from rare earth elements inclusive of Sc and Y), typically Nd-Fe-B sintered magnets, the inventors have found that when Dy and/or Tb and fluorine are absorbed and infiltrated in the magnet from its surface, Dy and/or Tb and fluorine are enriched only in proximity to interfaces between grains to impart a graded function that the coercive force becomes higher in the surface layer than in the interior, and especially the coercive force increases from the interior toward the surface layer. As a consequence, heat resistance is efficiently improved.

[0010] Accordingly, the present invention provides a functionally graded rare earth permanent magnet in the form of a sintered magnet body having an alloy composition  $R^1_a R^2_b T_c A_d F_e O_f M_g$  wherein  $R^1$  is at least one element selected from rare earth elements inclusive of Sc and Y and exclusive of Tb and Dy,  $R^2$  is one or both of Tb and Dy, T is one or both of iron and cobalt, A is one or both of boron and carbon, F is fluorine, O is oxygen, and M is at least one element selected from the group consisting of Al, Cu, Zn, In, Si, P, S, Ti, V, Cr, Mn, Ni, Ga, Ge, Zr, Nb, Mo, Pd, Ag, Cd, Sn, Sb, Hf, Ta, and W, a through g indicative of atom percents of the corresponding elements in the alloy have values in the range:  $10 \leq a+b \leq 15$ ,  $3 \leq d \leq 15$ ,  $0.01 \leq e \leq 4$ ,  $0.04 \leq f \leq 4$ ,  $0.01 \leq g \leq 11$ , the balance being c, said magnet body having a center and a surface. Grain boundaries surround primary phase grains of  $(R^1, R^2)_2 T_{14} A$  tetragonal system within the sintered magnet body. The concentration of  $R^2/(R^1+R^2)$  contained in the grain boundaries is on the average higher than the concentration of  $R^2/(R^1+R^2)$  contained in the primary phase grains.  $R^2$  is distributed such that its concentration increases on the average from the center toward the surface of the magnet body. The oxyfluoride of  $(R^1, R^2)$  is present at grain boundaries in a grain boundary region that extends from the magnet body surface to a depth of at least 20  $\mu m$ . The magnet body includes a surface layer having a higher coercive force than in the magnet body interior.

[0011] In a preferred embodiment, the oxyfluoride of  $(R^1, R^2)$  at grain boundaries contains Nd and/or Pr, and an atomic ratio of Nd and/or Pr to  $(R^1+R^2)$  contained in the oxyfluoride at grain boundaries is higher than an atomic ratio of Nd and/or Pr to  $(R^1+R^2)$  contained at grain boundaries excluding the oxyfluoride and the oxide of  $R^3$  wherein  $R^3$  is at least one element selected from rare earth elements inclusive of Sc and Y.

[0012] In preferred embodiments,  $R^1$  comprises at least 10 atom% of Nd and/or Pr; T comprises at least 60 atom% of iron; and A comprises at least 80 atom% of boron.

[0013] We find that permanent magnets as proposed herein can have a magnetic structure in which the coercive force of a surface layer is higher than in the interior, and efficiently improved heat resistance.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0014]

FIG. 1 is a graph in which the coercive force at varying sites of a magnet body M1 manufactured in Example 1 and a magnet body P1 as machined and heat treated is plotted relative to a depth from the magnet surface.

FIGS. 2a and 2b are photomicrographs showing Dy distribution images of the magnet bodies M1 and P1, respectively.

FIG. 3 is a graph in which the average concentrations of Dy and F in the magnet bodies M1 and P1 are plotted relative to a depth from the magnet surface.

FIG. 4a, 4b, and 4c are photomicrographs showing compositional distribution images of Nd, O, and F in the magnet body M1, respectively.

#### FURTHER EXPLANATIONS; OPTIONS AND PREFERENCES

[0015] The rare earth permanent magnet of the present invention is in the form of a sintered magnet body having an alloy composition of the formula (1).



Herein  $R^1$  is at least one element selected from rare earth elements inclusive of Sc and Y and exclusive of Tb and Dy,  $R^2$  is one or both of Tb and Dy, T is one or both of iron (Fe) and cobalt (Co), A is one or both of boron and carbon, F is fluorine, O is oxygen, and M is at least one element selected from the group consisting of Al, Cu, Zn, In, Si, P, S, Ti, V, Cr, Mn, Ni, Ga, Ge, Zr, Nb, Mo, Pd, Ag, Cd, Sn, Sb, Hf, Ta, and W. The subscripts a through g indicative of atom percents

well as the above-described casting technique.

[0028] The mother alloy is generally crushed to a size of 0.05 to 3 mm, preferably 0.05 to 1.5 mm. The crushing step uses a Brown mill or hydriding pulverization, with the hydriding pulverization being preferred for those alloys as strip cast. The coarse powder is then finely divided to a size of generally 0.2 to 30  $\mu\text{m}$ , preferably 0.5 to 20  $\mu\text{m}$ , for example, by a jet mill using nitrogen under pressure. The oxygen content of the sintered body can be controlled by admixing a minor amount of oxygen with the pressurized nitrogen at this point. The oxygen content of the final sintered body, which is given as the oxygen introduced during the preparation of the ingot plus the oxygen taken up during transition from the fine powder to the sintered body, is preferably 0.04 to 4 atom%, more preferably 0.04 to 3.5 atom%.

[0029] The fine powder is then compacted under a magnetic field on a compression molding machine and placed in a sintering furnace. Sintering is effected in vacuum or in an inert gas atmosphere usually at a temperature of 900 to 1,250°C, preferably 1,000 to 1,100°C. The thus sintered magnet contains 60 to 99 vol%, preferably 80 to 98 vol% of the tetragonal  $\text{R}_2\text{Fe}_{14}\text{B}$  compound as a primary phase, the balance being 0.5 to 20 vol% of an R-rich phase, 0 to 10 vol% of a B-rich phase, 0.1 to 10 vol% of R oxide, and at least one of carbides, nitrides and hydroxides of incidental impurities or a mixture or composite thereof.

[0030] The sintered block is machined into a magnet body of a predetermined shape, after which rare earth elements, typically Tb and/or Dy, and fluorine are absorbed and infiltrated in the magnet body in order to impart the characteristic magnetic structure that the coercive force of a surface layer is higher than in the interior.

[0031] Referring to a typical treatment, a powder containing Tb and/or Dy and fluorine atoms is disposed on the surface of the magnet body. The magnet body packed with the powder is heat treated in vacuum or in an atmosphere of inert gas such as Ar or He at a temperature of not higher than the sintering temperature (referred to as  $T_s$ ), preferably 200°C to  $(T_s-5)^\circ\text{C}$ , especially 250°C to  $(T_s-10)^\circ\text{C}$  for about 0.5 to 100 hours, preferably about 1 to 50 hours. Through the heat treatment, Tb and/or Dy and fluorine are infiltrated into the magnet from the surface and the rare earth oxide within the sintered magnet body reacts with fluorine to make a chemical change into an oxyfluoride.

[0032] The oxyfluoride of R (rare earth elements inclusive of Sc and Y) within the magnet is typically ROF, although it generally denotes oxyfluorides containing R, oxygen and fluorine that can achieve the effect of the invention including  $\text{RO}_m\text{F}_n$  (wherein m and n are positive numbers) and modified or stabilized forms of  $\text{RO}_m\text{F}_n$  wherein part of R is replaced by another metal element.

[0033] The amount of fluorine absorbed in the magnet body at this point varies with the composition and particle size of the powder used, the proportion of the powder occupying the magnet surface-surrounding space during the heat treatment, the specific surface area of the magnet, the temperature and time of the heat treatment although the absorbed fluorine amount is preferably 0.01 to 4 atom%, more preferably 0.05 to 3.5 atom%. From the standpoint of increasing the coercive force of a surface layer, it is further preferred that the absorbed fluorine amount be 0.1 to 3.5 atom%, especially 0.15 to 3.5 atom%. For absorption, fluorine is fed to the surface of the magnet body in an amount of preferably 0.03 to 30 mg/cm<sup>2</sup>, more preferably 0.15 to 15 mg/cm<sup>2</sup> of the surface.

[0034] Through the heat treatment, the Tb and/or Dy component also concentrates adjacent to the grain boundaries to augment anisotropy. The total amount of Tb and Dy absorbed in the magnet body is preferably 0.005 to 2 atom%, more preferably 0.01 to 2 atom%, even more preferably 0.02 to 1.5 atom%. For absorption, Tb and Dy are fed to the surface of the magnet body in a total amount of preferably 0.07 to 70 mg/cm<sup>2</sup>, more preferably 0.35 to 35 mg/cm<sup>2</sup> of the surface.

[0035] The surface layer of the magnet body thus obtained has a coercive force which is higher than the coercive force of the magnet interior. Although the difference in coercive force between the surface layer and the interior is not critical, the fact that the permeance differs about 0.5 to 30% between the surface layer and the interior suggests that the coercive force of the surface layer should preferably be higher than the coercive force of the magnet body interior (that is disposed at a depth of at least 2 mm from the magnet body surface) by 5 to 150%, more preferably 10 to 150%, even more preferably 20 to 150%.

[0036] It is understood that the coercive force of different sites in the magnet body can be determined by cutting the magnet body into discrete small pieces and measuring the magnetic properties of the pieces.

[0037] The permanent magnet material of the invention has a graded function that the coercive force of a surface layer is higher than that of an interior and can be used as a permanent magnet having improved heat resistance, especially in applications including motors and pickup actuators.

#### EXAMPLE

[0038] Examples of the present invention are given below by way of illustration and not by way of limitation.

#### Example 1 and Comparative Example 1

[0039] An alloy in thin plate form was prepared by using Nd, Cu, Al, and Fe metals of at least 99 wt% purity and



with an Ar atmosphere where it was sintered at 1,060°C for 2 hours, obtaining a magnet block. The magnet block was machined on all the surfaces into a disk having a diameter of 10 mm and a thickness (oriented direction) of 7 mm. This magnet body had an average permeance value of 2. The magnet body was successively washed with alkaline solution, deionized water, aqueous nitric acid and deionized water, and dried.

[0049] Next, terbium fluoride powder having an average particle size of 10  $\mu\text{m}$  was dispersed in deionized water in a mixing proportion of 50 wt%. The magnet body was immersed in the dispersion for 1 minute while sonicating the dispersion at 48 kHz, taken up and immediately dried with hot air. The amount of terbium fluoride fed was 1.2 mg/cm<sup>2</sup>. Thereafter, the packed magnet body was subjected to absorptive treatment in an Ar atmosphere at 800°C for 5 hours and then aging treatment at 510°C for 1 hour and quenched, obtaining a magnet body within the scope of the invention. This magnet body is designated M2. For comparison purposes, a magnet body was similarly prepared by effecting heat treatment without the terbium fluoride package. This is designated P2.

[0050]: The magnet bodies M2 and P2 were measured for magnetic properties (Br, H<sub>cj</sub>) and the maximum service temperature as defined in Example 1, with the results shown in Table 1. The compositions of the magnets are shown in Table 2. As compared with the magnet P2, the magnet M2 of the invention exhibited a substantially equal remanence, a high coercive force and a maximum service temperature rise of 45°C. The distributions of Tb and F in the magnet bodies M2 and P2 as analyzed by EPMA were equivalent to the distributions of Dy and F in Example 1. The distribution of coercive force of small pieces cut out of the magnet was the same as in Example 1.

[0051] These data prove that a magnet body characterized by the enrichment of Tb at grain boundaries, the dispersion of oxyfluoride, the graded concentrations of Tb and F, and the distribution of coercive force in the interior exhibits better heat resistance with a minimal amount of Tb added.

#### Examples 3-7 and Comparative Examples 3-7

[0052] An alloy in thin plate form was prepared by using Nd, Pr, Dy, Al, Fe, Cu, Co, Ni, Mo, Zr, and Ti metals of at least 99 wt% purity and ferroboron, weighing predetermined amounts of them, high-frequency melting them in an Ar atmosphere, and casting the melt onto a single chill roll of copper (strip casting technique). The alloy consisted of 11.5 atom% Nd, 1.0 atom% Pr, 1.0 atom% Dy, 0.5 atom% Al, 0.3 atom% Cu, 1.0 atom% M' (= Cr, Ni, Mo, Zr or Ti), 5.8 atom% B, and the balance of Fe.

[0053] The alloy was ground to a size of under 30 mesh by the hydriding technique. On a jet mill using nitrogen gas under pressure, the coarse powder was finely divided into a powder with a mass base median diameter of 5.1  $\mu\text{m}$ . The fine powder was oriented in a magnetic field of 15 kOe under a nitrogen atmosphere and compacted under a pressure of about 1 ton/cm<sup>2</sup>. The compact was then transferred to a sintering furnace with an Ar atmosphere where it was sintered at 1,060°C for 2 hours, obtaining a magnet block. The magnet block was machined on all the surfaces into a disk having a diameter of 10 mm and a thickness (oriented direction) of 7 mm. This magnet body had an average permeance value of 2. The magnet body was successively washed with alkaline solution, deionized water, aqueous nitric acid and deionized water, and dried.

[0054] Subsequently the magnet body was immersed in a dispersion of 50 wt% a 90:10 (weight ratio) terbium fluoride/neodymium oxide powder mix in ethanol for 1 minute while sonicating the dispersion at 48 kHz. The terbium fluoride and neodymium oxide powders had an average particle size of 10  $\mu\text{m}$  and 1  $\mu\text{m}$ , respectively. The magnet was taken up and placed in a vacuum desiccator where it was dried at room temperature for 30 minutes while evacuating by a rotary pump. The amount of terbium fluoride fed was 1.5 to 2.3 mg/cm<sup>2</sup>. Thereafter, the packed magnet body was subjected to absorptive treatment in an Ar atmosphere at 900°C for 3 hours and then aging treatment at 500°C for 1 hour and quenched, obtaining a magnet body within the scope of the invention. These magnet bodies are designated M3 to M7 in the order of M' = Cr, Ni, Mo, Zr, and Ti. For comparison purposes, magnet bodies were similarly prepared by effecting heat treatment without the powder package. They are designated P3 to P7.

[0055] The magnet bodies M3 to M7 and P3 to P7 were measured for magnetic properties (Br, H<sub>cj</sub>) and the maximum service temperature as defined in Example 1, with the results shown in Table 1. The compositions of the magnets are shown in Table 2. As compared with the comparative magnets, the magnets M3 to M7 of the invention exhibited substantially equal magnetic properties and a maximum service temperature rise of 20-30°C. The distributions of Tb and F in the magnet bodies M3 to M7 and P3 to P7 as analyzed by EPMA were equivalent to the distributions of Dy and F in Example 1. The distribution of coercive force of small pieces cut out of each magnet was the same as in Example 1.

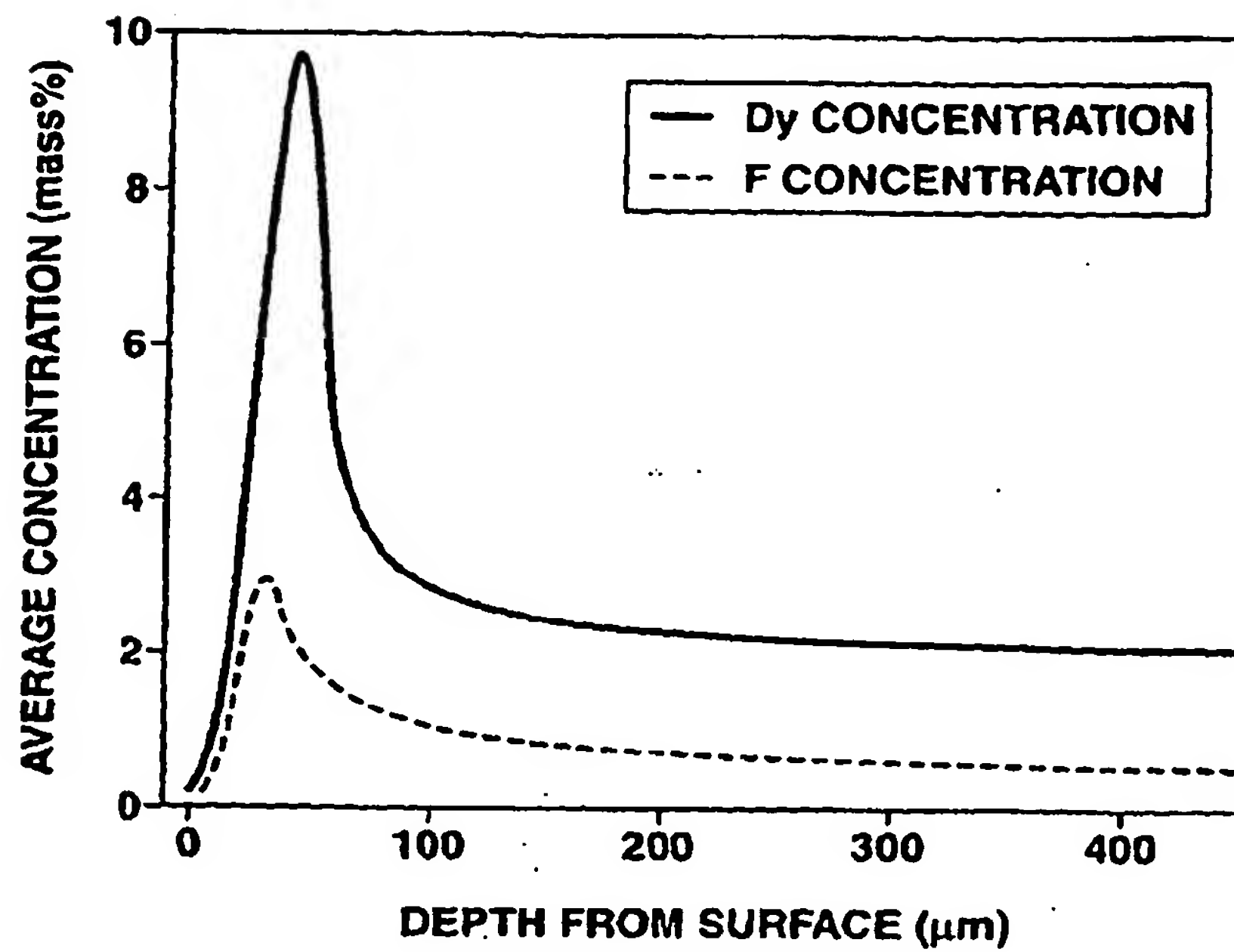
[0056] These data prove that a magnet body characterized by the enrichment of Tb at grain boundaries, the dispersion of oxyfluoride, the graded concentrations of Tb and F, and the distribution of coercive force in the interior exhibits better heat resistance with a minimal amount of Tb added.

- amples and Comparative Examples) in aqua regia, and effecting measurement by inductively coupled plasma (ICP), analytical values of oxygen determined by inert gas fusion/infrared absorption spectroscopy, and analytical values of fluorine determined by steam distillation/Alfusone colorimetry.

[0058] It will be understood that in numerical ranges herein, where the technical reasons for the upper limits naturally differ from those for the lower limits, the upper and lower limits constitute independent technical criteria.

## Claims

1. A functionally graded rare earth permanent magnet in the form of a sintered magnet body having an alloy composition  $R^1_a R^2_b T_c A_d F_e O_f M_g$  wherein  $R^1$  is at least one element selected from rare earth elements, Sc and Y, but excluding Tb and Dy,  $R^2$  is one or both of Tb and Dy, T is one or both of iron and cobalt, A is one or both of boron and carbon, F is fluorine, O is oxygen, and M is at least one element selected from Al, Cu, Zn, In, Si, P, S, Ti, V, Cr, Mn, Ni, Ga, Ge, Zr, Nb, Mo, Pd, Ag, Cd, Sn, Sb, Hf, Ta, and W, and indices a to g, indicating atom percents of the corresponding elements in the alloy have values satisfying:  $10 \leq a+b \leq 15$ ,  $3 \leq d \leq 15$ ,  $0.01 \leq e \leq 4$ ,  $0.04 \leq f \leq 4$ ,  $0.01 \leq g \leq 11$ , the balance being c, said magnet body having a center and a surface, wherein grain boundaries surround primary phase grains of  $(R^1, R^2)_2 T_{14} A$  tetragonal system within the sintered magnet body, the  $R^2$  concentration  $R^2 / (R^1 + R^2)$  contained in the grain boundaries is on the average higher than the  $R^2$  concentration  $R^2 / (R^1 + R^2)$  contained in the primary phase grains,  $R^2$  is distributed such that its concentration increases on average from the center toward the surface of the magnet body, the oxyfluoride of  $(R^1, R^2)$  is present at grain boundaries in a grain boundary region that extends from the magnet body surface to a depth of at least 20  $\mu m$ , and the magnet body includes a surface layer having a higher coercive force than the magnet body interior.
2. The rare earth permanent magnet of claim 1 wherein the oxyfluoride of  $(R^1, R^2)$  at grain boundaries contains Nd and/or Pr, and an atomic ratio of Nd and/or Pr to  $(R^1 + R^2)$  contained in the oxyfluoride at grain boundaries is higher than an atomic ratio of Nd and/or Pr to  $(R^1 + R^2)$  contained at grain boundaries excluding the oxyfluoride and the oxide of  $R^3$  wherein  $R^3$  is at least one element selected from rare earth elements inclusive of Sc and Y.
3. The rare earth permanent magnet of claim 1 or 2 wherein  $R^1$  comprises at least 10 atom% of Nd and/or Pr.
4. The rare earth permanent magnet of any one of claims 1 to 3 wherein T comprises at least 60 atom% of iron.
5. The rare earth permanent magnet of any one of claims 1 to 4 wherein A comprises at least 80 atom% of boron.
6. A method of making a rare earth permanent magnet having a surface layer with higher coercive force than the interior, as defined in claim 1, comprising providing an R-Fe-B sintered magnet body and causing Tb and/or Dy and fluorine to be absorbed and infiltrated into the body from its surface.

**FIG.3****FIG.4**

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the center toward the surface of the magnet body, the oxyfluoride of  $(R^1, R^2)$  is present at grain boundaries in a grain boundary region that extends from the magnet body surface to a depth of at least 20  $\mu m$ , and the magnet body includes a surface layer having a higher coercive force than in the interior. The invention provides permanent magnets having improved heat resistance.

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**ANNEX TO THE EUROPEAN SEARCH REPORT  
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